

# Y-12

**OAK RIDGE  
Y-12  
PLANT**

**LOCKHEED MARTIN** 

MANAGED BY  
LOCKHEED MARTIN ENERGY SYSTEMS, INC.  
FOR THE UNITED STATES  
DEPARTMENT OF ENERGY

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Revision 2**

## **Chemical Reactivity Testing for the National Spent Nuclear Fuel Program**

### **Quality Assurance Project Plan**

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Lockheed Martin Energy Systems, Inc.

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Machine Kinetics Corporation

February 8, 2000

Prepared by the  
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U.S. DEPARTMENT OF ENERGY  
under contract DE-AC05-84OR2140

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**CHEMICAL REACTIVITY TESTING FOR THE NATIONAL SPENT NUCLEAR FUEL  
PROGRAM**

**QUALITY ASSURANCE PROJECT PLAN**

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## CONTENTS

1.0 INTRODUCTION	1
1.1 Project Description and Objectives	1
1.1.1 Passivation of Uranium Powder With Oxygen and Water	1
1.1.2 Passivation of Uranium Powder Surface Characterization	1
1.1.3 Electrochemical Measurement of Uranium Hydride Corrosion Rate	1
2.0 ORGANIZATION	2
3.0 QUALITY ASSURANCE PROGRAM	2
3.1 General	2
3.2 Planning	3
3.3 Surveillances	3
3.4 Document Review	3
3.5 Personnel Selection, Indoctrination, and Training	4
3.6 Stop Work Authority	4
4.0 PROCUREMENT DOCUMENT CONTROL	5
5.0 IMPLEMENTING DOCUMENTS	5
6.0 DOCUMENT CONTROL	6
7.0 CONTROL OF PURCHASED ITEMS AND SERVICES	7
8.0 CONTROL OF MEASURING AND TEST EQUIPMENT	7
9.0 CORRECTIVE ACTION	15
10.0 QUALITY ASSURANCE RECORDS	15
11.0 AUDITS	15
12.0 SOFTWARE	16
13.0 SAMPLE CONTROL	16
14.0 SCIENTIFIC INVESTIGATION	16
15.0 CONTROL OF ANALYTICAL SERVICES	17
APPENDIX A, PROJECT ORGANIZATION	19
APPENDIX B, DEFINITION OF WORK SCOPE AND OBJECTIVES	20
APPENDIX C, DESCRIPTION OF THE SCIENTIFIC APPROACH AND METHODOLOGY THAT WILL BE USED	21
APPENDIX D, APPLICABLE PROCEDURES	25

APPENDIX E, EQUIPMENT AND LABORATORY APPARATUS	26
APPENDIX F, METHODS OF DOCUMENTING DATA	28
APPENDIX G, CONTROL AND STORAGE OF RECORDS	29
APPENDIX H, QUALITY ASSURANCE VERIFICATION OF THE WORK	30
APPENDIX I, PREREQUISITES	31
APPENDIX J, SPECIAL CONTROLS	32
APPENDIX K, SOFTWARE	34
APPENDIX L, JOB DESCRIPTIONS FOR MAJOR PROJECT PARTICIPANTS	36

## **LIST OF ACRONYMS**

ASO	Analytical Services Organization
BMPs	Best Management Practices
CN	Communication Notice
DCN	Document Change Notice
DOE	Department of Energy
DRIFT	Diffuse Reflective Fourier Transform Infrared
EIFT	Emission Fourier Transform Infrared
FTIR	Fourier Transform Infrared
LMES	Lockheed Martin Energy Systems, Incorporated
LMERC	Lockheed Martin Energy Research Corporation
M&TE	Measurement and Test Equipment
MMES	Martin Marietta Energy Systems, Incorporated
NIST	National Institute of Standards and Technology
NSNFP	National Spent Nuclear Fuel Program
OCRWM	Office of Civilian Radioactive Waste Management
OR	Oak Ridge
PI	Principal Investigator
QA	Quality Assurance
QAPjP	Quality Assurance Project Plan
QARD	Quality Assurance Requirements and Description

## 1.0 INTRODUCTION

This quality assurance project plan (QAPjP) summarizes requirements used by Lockheed Martin Energy Systems, Incorporated (LMES) Development Division at Y-12 for conducting chemical reactivity testing of Department of Energy (DOE) owned spent nuclear fuel, sponsored by the National Spent Nuclear Fuel Program (NSNFP). The requirements are based on the NSNFP Statement of Work PRO-007 (Statement of Work for Laboratory Determination of Uranium Hydride Oxidation Reaction Kinetics.)

This QAPjP will utilize the quality assurance program at Y-12, Y60-101PD, *Quality Program Description*, and existing implementing procedures for the most part in meeting the NSNFP Statement of Work PRO-007 requirements, exceptions will be noted.

### 1.1 Project Description and Objectives

The project consists of conducting three separate series of related experiments, “Passivation of Uranium Hydride Powder With Oxygen and Water”, “Passivation of Uranium Hydride Powder with Surface Characterization”, and “Electrochemical Measurement of Uranium Hydride Corrosion Rate”.

#### 1.1.1 Passivation of Uranium Powder With Oxygen and Water

The basic passivation mechanism of uranium hydride will be measured by very gently titrating the surface of uranium hydride powder a fraction of a monolayer at a time. This will be followed by subsequent oxidation at higher oxygen activities to determine the rates and mechanisms of purely oxidative passivation by monitoring oxygen consumption and water production. The first objective will be to observe the rate at which the fresh surface is converted from hydride to oxide by oxygen, and the production of water. The second objective will be to observe the rate at which oxygen reacts with this slightly passivated surface to develop more passivation and more tolerance for the oxygen. The reaction extent will be measured by the hydrogen pressure rise in the presence of the water vapor at constant pressure. Initial experiments will be carried out at ambient temperature.

#### 1.1.2 Passivation of Uranium Powder Surface Characterization

Uranium will be hydrided inside an evacuable cell such that the uranium hydride can be monitored by Diffuse Reflectance Fourier Transform Infrared (DRIFT) Spectroscopy, or by Emission Fourier Transform Infrared (EFT) Spectroscopy. DRIFT will measure the growth of uranium oxides and EFT will measure thermal emissivity. This will allow the measurement of the oxidation kinetics in the solid phase of the material, rather than measuring gas phase reactions products under the experiment described in 1.1.1.

#### 1.1.3 Electrochemical Measurement of Uranium Hydride Corrosion Rate

This approach involves the measurement, using standard computer-controlled electrochemical hardware and appropriate control and data acquisition/analysis software, of the rate of corrosion of uranium hydride surfaces. The hydride surfaces will be prepared by either in-situ electrochemical generation of hydride films on mounted depleted uranium specimens, or by polishing of mounted and compacted  $\text{UH}_3$  material. Material preparation will be clearly documented for each experiment. The testing scheme will use a range of water and oxygen concentrations up to and including pure water and pure oxygen, ranging downward as feasible to



determine the relationship of the measured corrosion rate to concentration of these components of the environment. Results from these experiments are expected to be connectable to the results of the gas-phase experiments, to extend the understanding of the corrosion phenomenon beyond what would be possible with either technique alone.

## **2.0 ORGANIZATION**

The LMES Development Division at Y-12 will be the responsible organization for conducting this project. Roles and responsibilities are illustrated via the organization chart in Appendix A and as follows:

G. Louis Powell: Overall Project Manager reporting to P. E. McKenzie, Group Leader for the Compatibility and Certification Group of the Y-12 Development Division, also serving as the Principal Investigator for the Passivation of Uranium Powder with Oxygen and Water and the Passivation of Uranium Powder Surface Characterization experiments using the gas burette techniques and FTIR spectroscopy.

Jonathan S. Bullock IV: Principal Investigator for Electrochemical Measurement of Uranium Hydride Corrosion Rate Experiment. Reporting to the Project Manager.

Louis W. Koester: Quality Engineer for the project, responsible for ensuring the project activities comply with the quality requirements of the NSNFP Statement of Work, PRO-007 and/or identifying and documenting any exceptions. Reporting to P. E. McKenzie and Y-12 Quality Manager. Louis Koester will replace Hugh Newsom as the Quality Engineer as of January 31, 2000

## **3.0 QUALITY ASSURANCE PROGRAM**

### **3.1 General**

The approval signature on the Review and Approval Sheet of this QAPjP by the Compatibility and Certification Group Leader person signifies that all personnel associated with this project are directed to comply with all the requirements of this project plan, with no exceptions, unless reviewed by the Quality Engineer and approved by Compatibility and Certification Group Leader.

The existing QA program for Y-12, Y60-101PD, *Quality Program Description*, shall be used along with the existing implementing procedures to translate the NSNFP Statement of Work PRO-007 requirements into work processes. Where no existing procedure exists, the implementing process shall be described within this plan.

### **3.2 Planning**

General project planning is contained within this plan and will address a) through m) below as a minimum. This information will be contained in appendices correlated to a) through m), with noted exceptions. Experiment-specific planning issues not contained in the general project planning and/or changes shall be documented by Document Change Notices (DCNs) (reference Section 6.0) and shall be each PI's responsibility.

- a) Definition of work scope and objectives, reference Appendix B;

- b) Description of the scientific approach and methodology that will be used, reference Appendix C;
- c) Applicable codes and standards, not applicable at this time;
- d) Y-12 procedures that will be applicable, reference Appendix D;
- e) Equipment and laboratory apparatus, reference Appendix E;
- f) Methods of documenting data, reference Appendix F;
- g) Control and storage of records, reference Appendix G;
- h) Quality Assurance verification of the work, reference Appendix H;
- i) Prerequisites, reference Appendix I;
- j) Special controls, reference Appendix J;
- k) Environments or skills, not applicable at this time;
- l) Software, reference Appendix K; and
- m) Job descriptions for major project participants, reference Appendix L.

All activity plans, experiment plans, and revisions shall be submitted to NSNFP for concurrence prior to implementation.

### **3.3 Surveillances**

The Project Quality Engineer will conduct surveillances of the work in accordance with Y-12 procedure Y60-904, *Surveillances*, Sections B, C, and D. A surveillance of each chemical reactivity experiment/test will be conducted during the duration of the experiment/test as a minimum. Each surveillance will be documented in a report to the responsible Project Manager. Deficiencies identified during surveillances are to be handled in accordance with the Y-12 corrective action program, reference procedure Y60-312, *Issues Management Program*.

### **3.4 Document Review**

All implementing documents shall reflect evidence of a review, including this QAPjP, by qualified individuals other than the preparer. All experiment plans and revisions thereto shall be submitted for review by NSNFP.

The review and approval process for all implementing documents, which includes DCNs that change and/or modify this QAPjP, shall be as follows:

- a) The reviewer list shall consist of the PIs, Project Manager, the Project Quality Engineer, as a minimum. Additional reviewers may be selected based on applicable technical expertise. The preparer cannot participate as a reviewer.

- b) The reviewers shall categorize any comments made into two categories:
  - 1) mandatory
  - 2) nonmandatory
- c) Comments are documented and forwarded to the preparer or person responsible for circulating the document. Comments shall be transmitted by E-mail except in cases where hard copies are mandated by signature requirements or physical limitations of E-mail such as file size.
- d) Mandatory comments must be considered for inclusion into the document being reviewed. The resolution of mandatory comments shall be concurred with by the originating reviewer and documented, or elevated to the Project Manager for resolution.
- e) Nonmandatory comments are considered for inclusion at the preparer's discretion.
- f) The Project Manager shall be responsible for mediating any dispute that arises between the reviewer and preparer over inclusion of mandatory comments. The resolution shall be documented.
- g) Mandatory and nonmandatory comments as well as "No Comment" responses shall be retained as project records and filed in correlation to the related document.
- h) The approval process shall require the Y-12 Development Division Compatibility and Certification Group Leader signature.

### **3.5 Personnel Selection, Indoctrination, and Training**

Job descriptions for the major project positions shall be established and defined, reference Appendix L.

Personnel actively involved with this project shall have experience, education, and training commensurate with the project scope.

Education and experience shall be verified and documented via Human Resources and the LMES Personnel Organization. Where this cannot be accomplished, the reason(s) and alternative methods used by the Project Manager in completing the process shall be documented. Experience evaluation shall be by review of each persons resumé and/or knowledge of past and present assignments by the Project Manager and documented. Resumés of project personnel shall be maintained as project records.

Personnel shall receive indoctrination and training in the requirements of this QAPjP and the implementing documents the project work will be done under. Records of training shall be maintained as project records.

### **3.6 Stop Work Authority**

Stop Work Authority resides in the Project Manager, PIs, and the Quality Engineer and is ultimately upheld by line management. The implementing procedure for this process is Y60-551, *Stop Work/Restart Authority*.

#### **4.0 PROCUREMENT DOCUMENT CONTROL**

Procurement documents shall be prepared for all quality affecting items and services obtained from suppliers external and internal to Y-12. Procurement documents shall include technical and quality assurance requirements necessary to ensure quality and be reviewed by the Quality Engineer before being issued. When items are being procured which includes initial calibration or calibration services, the calibration data sheet, calibration certificate and statement of traceability shall be required to be furnished by the vendor.

Project procurement will be done in accordance with the Y-12 procedure Y60-701, *Procurement Quality*, Section L, "Commercial Grade Items".

Suppliers furnishing items and/or services deemed critical to the success of this project shall be evaluated as to their capability of performance based on the following criteria:

- a) Does the supplier have an ISO Program in place such as ISO 9001, or ISO 9002?
- b) Does the supplier have a Quality Assurance Program implemented such as NQA-1, DOE Order 414.1 (superseding 5700.6C), or OCRWM's QARD?
- c) Has the supplier been used in the past?
- d) If used in the past, were the services and/or items furnished acceptable and delivered on time?
- e) Has this supplier been assessed by DOE or another DOE contractor/subcontractor? Did the assessment indicate a Quality Program had been effectively implemented, or as a minimum was the supplier declared "acceptable"?

A supplier must receive a positive evaluation based on two of the criteria (a through e) as a minimum, to be placed on the approved suppliers list. The Project Manager and the Project Quality Engineer must approve any variation from this requirement with justification documented.

The suppliers shall be required to allow access to their facilities and records, by project personnel and/or NSNFP representatives when deemed necessary.

#### **5.0 IMPLEMENTING DOCUMENTS**

Work and activities affecting the project work shall be accomplished in accordance with the Y-12 QA Program as detailed in, Y60-101PD *Quality Program Description*, this QAPjP and Y-12 implementing procedures. Any additional required instructions will be generated as Document Change Notices and/or Communication Notices with a review and approval process. (Reference Section 3.4.)

Implementing documents shall be in place for the following activities as a minimum:

- a) All experimental activities;
- b) Internal M&TE calibration;

- c) Chemical analysis; and
- d) Supplier qualification.

## **6.0 DOCUMENT CONTROL**

The originating organization will be responsible for document control of the Y-12 Site documents. Documents generated by the project that are to be controlled shall be prepared, reviewed, approved and issued in accordance with this section of the QAPjP, and procedure Y15-102, *Document Control*. Documents and changes thereto will be reviewed prior to issue for technical adequacy, completeness, correctness, inclusion of appropriate QA requirements, and distribution. The project will be responsible for controlling and distributing the following documents:

- a) This Quality Assurance Project Plan (QAPjP);
- b) Document Change Notices (DCNs) generated and issued by the project; and
- c) Communication Notices (CNs) generated and issued by the project.

### **Quality Assurance Project Plan**

Preparation Responsibility.... Project Quality Engineer

Review Responsibility..... Project Principal Investigators, Y-12 QA Manager

Approval Responsibility..... Group Leader for the Compatibility and Certification Group

### **Document Change Notice (DCNs)**

DCNs shall be a process to communicate document changes and/or additions that need to be made within a short time frame. Planning documents may utilize this process. DCNs may be transmitted via E-mail, hard copy and/or FAX. The numbering process will begin with DCN-01, and will be assigned by the Project Quality Engineer. Each DCN shall be reviewed by the Quality Engineer before being released and shall state the effective date. DCNs shall become part of the project records.

Preparation Responsibility..... Project Personnel

Review Responsibility..... Determined by the Creator, Quality Engineer as a minimum

Approval Responsibility..... Project Manager

Maintenance of DCN Log..... Project Support/Project Quality Engineer

Distribution Responsibility..... Creator

The Project Manager and the Quality Engineer shall be on the distribution list of all DCNs as a minimum. DCNs that change or modify this QAPjP shall be subjected to a review and approval process described in Section 3.4. Determination of distribution will be made as to the personnel and/or organizations impacted and original document distribution if in existence. Semiannually, all DCNs issued will be reviewed by the Quality Engineer to determine if the document changes/modifications transmitted by this process warrants revising this QAPjP.

## **Communication Notice (CNs)**

CNs shall be a process to document communications of a general nature or as a cover letter to transmit technical information. The CN process will be used for tracking corrective actions to closure and any related communication requirements. The numbering process will begin with CN-01 and will be assigned by the Project Quality Engineer. CNs shall become part of the project records.

Preparation Responsibility..... Project Personnel  
Review Responsibility..... Determined by the Creator  
Approval Responsibility..... Determined by the Creator  
Maintenance of CN Log..... Project Support/Project Quality Engineer  
Distribution Responsibility..... Creator

## **7.0 CONTROL OF PURCHASED ITEMS AND SERVICES**

The project will use the existing process in place at LMES, procedure Y60-701, *Procurement Quality*, Section J, “Acceptance of Items and Services; and Section L, “Commercial Grade Items”. Additionally the Principal Investigators of the project shall be responsible for receipt, inspection, acceptance, and control of items and/or documents furnished by suppliers. Documents include specification and calibration records of procured equipment used in the experiments. The results of the receipt inspection and acceptance process shall be documented.

The specification and calibration records shall be filed in a manner that allows easy access for project audits and surveillances.

Nonconformances noted at the receipt, inspection, or checkout phases shall be identified and controlled via Y-12 Procedure Y60-301, *Control of Nonconforming Items (and Services)*. The Project Quality Engineer shall be notified. The CN process shall be used to further document this process.

## **8.0 CONTROL OF MEASURING AND TEST EQUIPMENT**

The project will utilize the existing Y-12 program and implementing procedure Y60-802, *Calibration and Control of Measuring and Test Equipment (M&TE)*, Section B, “Control of M&TE”. This procedure shall be the basis for the control of the project M&TE.

The project PIs shall be responsible for the following actions:

- a) Identifying M&TE used for each experiment and/or test that controls or measures any of the parameters;
- b) Establishing the method and interval of calibration for each device identified in a), based on the type of equipment, intended use, required accuracy and other conditions affecting measurement control;

- c) Ensuring that when calibrations are made against standards, the standard has a greater accuracy than that of the required accuracy of the equipment being calibrated;
- d) Ensuring that when M&TE is calibrated against reference standards, they are traceable to nationally recognized standards such as NIST, or documented justification for variations;
- e) Documenting the M&TE used for each experiment to allow for the evaluation of data should the equipment be found to be out of calibration after completion of the experiment;
- f) Ensuring that when M&TE is found to be out of calibration, the validity of the results obtained using that equipment since its last calibration are evaluated and documented;
- g) Uniquely identifying each calibrated M&TE item to provide traceability to its calibration data;
- h) Ensuring M&TE is labeled, tagged or appropriately documented to identify the due date or interval of the next calibration. Results of actions a) through h) shall be documented as part of this plan. Later changes and/or modifications shall be communicated via the DCN process;
- i) Ensuring the identity of the person performing each calibration is known and documented;
- j) Ensuring the recorded calibration data includes for each calibration point the following as a minimum:
  - 1) required value
  - 2) tolerance
  - 3) as found condition
  - 4) calibrated value
- k) Internal calibration procedures shall be generated for each type of calibration and/or equipment, and implemented via the DCN process.

**Laboratory M&TE**

**Principal Investigator**

**G. Louis Powell**

Parameters to be measured are:

Pressure	Torr
Volume	Cubic centimeters
Temperature	Degrees C
Weight	Grams
Fourier Transform Infrared Spectra	Gas, DRIFT, EIFT
Voltage	Volts
Mass Spectrometer	H
Gases	H <sub>2</sub> , O <sub>2</sub> , H <sub>2</sub> O
Time	Second, minutes, hours

Hardware to be used in measuring and/or controlling the parameters, method of calibration, calibration intervals, and traceable standards where applicable are as follows:

### **(A) Pressure Calibration**

1. MKS-Baratron Pressure gauges (digital display and analog output)
  - a) Method: direct comparison
  - b) Interval: 2-year
  - c) Standard: similar NIST traceable MKS gauges
  - d) Required Accuracy: 1.0 % Full Scale
2. MKS Digital Meter: Provides a, voltage output correlated to the pressure gauge reading. A full scale setting from the Baratron pressure gauge registers a 10.000 reading on the digital meter, likewise 0 full scale equates to 0.000 reading.
  - a) Method: Correlation to the pressure gauge. The meter “zero” is checked at a  $>1 \times 10^{-5}$  torr vacuum with the Nicolet oscilloscope reading recorded to later offset zero.
  - b) Interval: beginning of each experiment
  - c) Standard: internal alignment
  - d) Required Accuracy:  $\pm 0.005$  digital display
3. Nicolet Oscilloscope: The MKS Digital Meter voltage output is fed to the “Y” axis of the oscilloscope.
  - a) Method: Selector switch on the front panel of the digital meter in allows a reading of 0.0 and 10.000 to be substituted for the pressure signal which allows for the oscilloscope’s calibration relative to the digital output. 0 and 10 volts (from 2.) are recorded in the notebook.
  - b) Interval: Per each experiment
  - c) Standard: pressure gauge full scale and null
  - d) Required Accuracy: 1%

The calibration of the pressure measuring system shall provide an absolute accuracy factor of 0.5% and shall be done at 2-year intervals.

### **(B) Volume**

- a) Method: Originally calibrated against an old internal standard by expansion. A new calibrated volume to be established using weighing before and after water filling.
- b) Interval: when the volume is modified
- c) Standard: stainless steel volume with valve, weighed dry and filled with water
- d) Required Accuracy: 1%

### **(C) Temperature**

1. Thermocouples (Omega) New type K, with digital readouts and analog output (OMEGA DP116-KC) to the Nicolet Oscilloscope.
  - a) Method: TCs, displays, & outputs individually certified by ORNL I&C calibration.
  - b) Interval: prior to installation
  - c) Standard: NIST traceable fluid bed comparison
  - d) Required Accuracy:  $\pm 2$  degree C



2. Temperature Control: Temperature control shall be by Harrick Scientific or Omega Engineering temperature controllers. Temperature control is not temperature measurement, and is not subject to calibration.

**(D) Weight**

1. Balances, Metler AE 163 (M-225137), Metler PE 360 (M-225136), and Sartorius MC1/LC 620-0 S(M-225138)
  - a) Method: weighing of standard weights
  - b) Interval: quarterly by Y-12 Balance Certification Program
  - c) Standard: NIST traceable weights
  - d) Required Accuracy:  $\pm 1$  mg,  $\pm 100$  mg for M-225138

**(E) Fourier Transform Infrared Spectra (Gas, DRIFT, EIFT)**

1. Bio-Rad FTIR FTS-60 #15050 w/Harrick Scientific Spectropus System ( DRIFT and EIFT)
  - a) Method: built into the instrument
  - b) Interval: before each experiment
  - c) Standard: X axis: helium neon laser  
Y axis: ratio to background
  - d) Required Accuracy: baseline, see items 1, 2, and 3
- 1) Wavenumber Calibration (X axis)
  - a) Method: done with internal He/Ne laser standard
  - b) Interval: beginning of experiment
  - c) Standard: He/Ne laser
  - d) Required Accuracy: 1 wavenumber
- 2) Absorbance/Reflectance (Y axis) Gas Cell
  - a) Method: ratio observation to reference spectrum gas.
  - b) Interval: every spectrum
  - c) Standard: reference spectrum open beam obtained through gas cell evacuated to  $<1 \times 10^{-5}$  torr
  - d) Required Accuracy: baseline width at  $2000 \text{ cm}^{-1} = 2 \times 10^{-3}$  absorbance units
- 3) DRIFT
  - a) Method: Uses sand blasted gold
  - b) Interval: per each experiment
  - c) Standard: sandblasted gold cleaned ultrasonically using 5% micro detergent plus ultrasonic distilled H<sub>2</sub>O rinse plus blow-dry
  - d) Required Accuracy: baseline width at  $2000 \text{ cm}^{-1} = 2 \times 10^{-1}$  absorbance units
- 4) EIFT
  - a) Method: Uses a “black body”, a graphite heated cylinder with a 3-mm hole 12 mm deep beginning the focal point of the collector
  - b) Interval: as needed
  - c) Standard: black body in a), at measured temperature
  - d) Required Accuracy: N/A

#### **(F) Voltage (Nicolet Oscilloscopes)**

- a) Method: On a service contract, with recalibration to NIST traceable voltage and frequency standards
- b) Interval: annually
- c) Standard: NIST traceable DC voltage and frequency standards, reported by Nicolet on calibration sheet
- d) Required Accuracy:  $\pm 1\%$

#### **(G) Mass Spectrometer (UTI 100C)**

- a) Method: Quantitative calibration is by the gas pulse method/mass calibration by gas leak
- b) Interval: before and after measurement pulse
- c) Standard: pressure and volume of gas before release
- d) Required Accuracy: N/A

#### **(H) Gases/Chemicals**

Hydrogen, oxygen, water and uranium will not be calibrated as M&TE equipment, however they remain as critical parameters to the experiments. Supplier certifications will be used to ensure the purity of hydrogen and oxygen. Lab analysis will be used to verify the uranium sample's chemical composition. Water analyses will be performed by the project. Certifications, project and lab analyses will become part of the project records.

#### **(I) Time**

##### 1. Absolute Time

- a) Method: NISTIMEW.exe on the Web per each experiment using the Bio-Rad computer
- b) Interval: beginning of each experiment
- c) Standard: time.a.nist.gov
- d) Required Accuracy: 0.1%

##### 2. Synchronization of Nicolet and Bio-Rad clocks

- a) Method: use time from Bio-Rad clock, for Nicolet time set
- b) Interval: beginning of each oscilloscope trigger
- c) Standard: time.a.nist.gov
- d) Required Accuracy: 0.1%

##### 3. Differential time (Bio-Rad/Nicolet)

- a) Method: calculated from time set of 1c and 2c documentation
- b) Interval: as necessary
- c) Standard: time.a.nist.gov
- d) Required Accuracy: 0.1%

Calibrations shall be carried out in accordance with procedures CRP-001 through CRP-013-refer to Appendix D.

Explicit calibration and interpretation of all spectrometric data are considered experimental results and the deduction of such are the outcome of research.

## **Laboratory M&TE**

### **Principal Investigator**

**Jon S. Bullock**

Parameters to be measured are:

Temperature	Degrees C
Pressure	Torr or Atmospheres
Flow	$\text{L s}^{-1} \text{ cm}^3 \text{ s}^{-1}$
Resistance/Capacitance	Ohms/Microfarads
pH	Dimensionless
Conductivity	$\text{Ohm}^{-1} \text{ cm}^{-1}$
Rotating Disk Electrode speed	Revolutions per minute or radians per second
Experiment Control System	Not applicable
Test Cell Reference Electrode	Volts or mV with respect to a specified reference electrode
Oxygen Content of Electrolyte	Molal

Hardware to be used in measuring and/or controlling the parameters and method of calibration are:

### **(A) Temperature**

1. Primary: Digital Process Indicator, Model DP81 for type T thermocouple, accuracy to 0.2 degrees K, Type T probes, Omega Engineering, Inc.
  - a) Method: ice point reference
  - b) Interval: monthly
  - c) Standard: ice point reference, Model K140-4A, S/N 16659, Kaye Instruments
  - d) Required Accuracy:  $\pm 1.0$  degrees C
2. Backup: Digital Thermometer, Model 871A for type K thermocouple, accuracy to 0.4 degrees K, S/Ns T-145216 and T-149171, Type K probes, Omega Engineering, Inc.

### **(B) Pressure**

1. Refer to G. Louis Powell's Laboratory M&TE Section of this plan.

### **(C) Flow**

1. Liquid: Peristaltic pump, accuracy of 0.25%
  - a) Method: volume displaced per unit time by direct measurement
  - b) Interval: monthly
  - c) Standard: graduated container
  - d) Required Accuracy: 1.0 %
2. Gas
  1. Rotameter, model E-03217-57, 2% accuracy, 0.25% repeatability, range approximately

1 cc/sec for argon and oxygen, Cole-Parmer, Inc.

a) Method: comparison to standard

b) Interval: annually unless evidence indicates higher frequency required

c) Standard: pre-standardized for 1 year; after that, by digital flowmeter, model E-32970-10, Cole-Parmer, Inc.

d) Required Accuracy: 1.0 %

#### **(D) Resistance**

1. Primary: Digital Micro-Ohmmeter, Valhalla Scientific, Model 4300B, S/N 32-714, ranges to 20,000 ohms

a) Method: direct measurement

b) Interval: bimonthly

c) Standard: General Radio Corporation Model 1433X, 0.02% accuracy

d) Required Accuracy: 0.5 %

2. Backup: Kiethley Instruments Model 197, S/N's 373369 and 373348

a) Method: direct measurement

b) Interval: bimonthly

c) Standard: General Radio Corporation Model 1433X, 0.02% accuracy

d) Required Accuracy: 0.5 %

#### **(E) Capacitance**

1. Capacitance Bridge, General Radio Corporation Model 1615-A, S/N 2248, Oak Ridge (OR) DOE property #Y23497

a) Method: direct measurement

b) Interval: bimonthly

c) Standard: fixed mica capacitor, General Radio Corporation Model 1409Y, nominal value 1.00 microfarad, accuracy to 0.05% of actual calibrated value, with certificate of calibration

d) Required Accuracy: 0.5 %

2. AC Impedance Dummy Cell, R-C circuit, series/parallel combination, contains capacitive elements of 100 microfarad value and resistive elements of 10 and 100 ohms. Model 1700-1126-Rev.0, EG&G S/N 001JSB

a) Method: direct measurement of R/C elements using resistance meter and capacitance bridge

b) Interval: bimonthly

c) Standard: fixed mica capacitor from General Radio Corp. Model 1409Y/decade resistor from General Radio Corporation Model 1433X

d) Required Accuracy: 0.75 %

#### **(F) pH Measurements**

1. Primary: pH Meter with ISFET Probe Model PHI 110, resolution to 0.001 pH unit, Beckman Instrument Co.

a) Method: calibration against standard solutions

b) Interval: weekly when experiments are in progress

c) Standard: standard solutions nominally within 2 pH units above and below the measured value of the experiment solutions

d) Required Accuracy: 0.1 unit

2. Backup: pH Meter with Standard Combination Probe Model PHI 34, resolution to 0.003 pH unit, Beckman Instrument Co.

#### **(G) Conductivity**

1. Alternate 1: Conductivity Meter w/glass-body cell, Model PCM3, S/N 2428, Jenway Co.
  - a) Method: adjustment of meter parameters to agree with standard solutions
  - b) Interval: biweekly while experiments are in progress
  - c) Standard: standard solution normally certified to 0.3% of accuracy
  - d) Required Accuracy: 1.0 %
- 2) Alternate 2: Specific Conductance Meter w/polymer-body cell, model 10054, S/N 9705006, VWR Scientific Inc. Conductivity standard solutions are 0.30% tolerance.

#### **(H) Rotating Disk Electrode Speed**

1. Rotating disk analyzer, rotating disk electrode w/ controller, Model AFMSRX, MSRS Analytical Rotator Speed Control, S/N 919, Pine Instrument Co.
  - a) Method: against an optical tachometer using a retro reflection principle
  - b) Interval: monthly
  - c) Standard: AC line frequency
  - e) Required Accuracy: 1.0 %

#### **(I) Experimental Control System Comprised of (1) and (2)**

1. Potentiostat/Galvanostat, Model 283, S/N 25101, EG&G Instruments
  - a) Method: measurement of dummy cell
  - b) Interval: biweekly
  - c) Standard: as transferred using dummy cell
  - d) Required Accuracy: 1 %, includes item 2)
2. Frequency Response Detector, Model 1025, S/N 12113, EG&G Instruments
  - a) Method: measurement of dummy cell
  - b) Interval: biweekly
  - c) Standard: as transferred using dummy cell
  - d) Required Accuracy: 1 % , includes item 1)

#### **(J) Test Cell Reference Electrode**

- a) Method: direct comparison of potential to standard
- b) Interval: daily
- c) Standard: Ag/AgCl reference electrode
- d) Required Accuracy: 0.005 volts

#### **(K) Oxygen Content of Electrolyte**

- a) Method: measurement of the oxygen-reduction limiting current on the platinum rotating disk electrode at a specified rotation speed
- b) Interval: each time the concentration is set to a new value
- c) Standard: rotating disk electrode
- d) Required Accuracy: 1 %

Calibrations shall be performed in accordance with procedures CRP-014 through CRP-025-refer to Appendix D.

Explicit calibration and interpretation of all electrochemical data are considered experimental results and the deduction of such are the outcome of research.

## **9.0 CORRECTIVE ACTION**

Conditions adverse to quality are to be identified and reported to the Project Manager for immediate action and to the Project Quality Engineer for tracking to closure. Responsible management shall determine the extent of the condition and complete remedial action as soon as practical. The Project Quality Engineer shall concur with the proposed remedial action to ensure QA program requirements are met. Conditions adverse to quality shall be evaluated to determine if stopping of work is warranted. Y-12 procedure, Y60-312, *Issues Management Program* shall be used in bringing about closure of corrective action items, with the exception of utilization of the Energy Systems Action Management System. Tracking and communication aspects related to corrective actions shall be documented via the Communication Notice (CN) process.

## **10.0 QUALITY ASSURANCE RECORDS**

Identification of quality assurance records by category is contained in Appendix G. In addition the planning and implementing documents will further identify documentation that will be considered quality assurance records. Corrections to completed and in-process quality assurance records shall be made by striking out the changed information with a single line and inserting new or corrected information with signature or initials of the person authorized to make the change and date change was made. All QA records shall be reviewed by the Quality Engineer for legibility and completeness. This review process shall be documented.

The Y-12 Development Division shall store all records in locked, one-hour fire-rated cabinets, or maintain dual storage facilities, with access controlled by the Project Manager. The records shall be indexed to ensure retrieveability until turned over to NSNFP. All documentation will eventually be converted to digital form and stored on CD-ROM.

## **11.0 AUDITS**

The project personnel will not conduct any internal audits, however internal surveillances will be planned and performed. The Development Division as a resident of the Y-12 Site will be subjected to periodic audits scheduled and conducted by organizations having overall compliance and QA responsibility on site. The project will provide access of facilities and records to NSNFP to allow acceptance and verification of the QA program and evaluation of the services being provided.

## 12.0 SOFTWARE

Software being used on this project is categorized into the following four categories:

- (A) Industry Standard;
- (B) Component of an equipment package for control or data acquisition;
- (C) Developed in house; and
- (D) Macros developed for (A) through (C).

Verification and validation of software in categories (C) and (D) shall be required before using. The two processes shall be implemented in accordance with Sections C3, G, H4, and I of procedure ESS-QA-19.0, *Software Quality Assurance*, and shall be documented, becoming part of the project records.

Software used in conducting each experiment and manipulation of data shall be identified including the version as part of the planning and results reporting process. Project software is listed in Appendix K.

## 13.0 SAMPLE CONTROL

Depleted uranium, the material for the project samples, will be handled and stored in accordance with the building radiological work permit covered by Y-12 procedure, Y75-122, *Radiological Work Permit*. Samples of depleted uranium, conditioned as part of the experiments are destroyed during the process, however any remaining residue will be returned to the depleted uranium or uranium hydride storage container. The size, volume in grams, thickness of the sample, shape, and characterization derived from the lab analysis will be recorded as part of the planning and results reporting process.

Plans are to transport as a minimum one sample to the Y-12 Laboratory for analysis as a typical composition. This sample container will carry appropriate identification and meet all site requirements contained in the Y-12 procedure Y75-101, *Transfer and Management of Material for Radiological Control*. The Analytical Chemistry Organization's chain-of-custody procedure, Y/P65-9094, *Chain-of-Custody in the Analytical Chemistry Organization*, and forms will be used to document the transfer of the sample custody to the Analytical Chemistry organization lab. A copy of the completed chain-of-custody form shall be maintained in the project records.

## 14.0 SCIENTIFIC INVESTIGATION

Scientific (lab) notebooks shall be used by each PI in preparing for and conducting each experiment. As a minimum the scientific notebook shall contain the following:

- a) Statement of objective;
- b) Description of the work to be performed;

- c) Reference to the work scope planning documents;
- d) Identification of individuals performing the research;
- e) Description of equipment, materials and apparatus;
- f) Identification of samples;
- g) M&TE calibration requirements;
- h) Description of the work as it is being performed;
- i) Results obtained;
- j) Dated initials or signatures of personnel making entries; and
- k) Description of any changes.

When information that falls into one of the categories (a through k) already exists in other documents, the scientific notebook entries need not repeat the information but provide a reference to the location.

The scientific notebooks shall be reviewed, as a minimum monthly, by an independent qualified individual to verify there is sufficient detail to retrace or repeat the investigation and to confirm the results without recourse to the original investigator. This requirement will be met by each project PI reviewing the other PI's scientific notebook.

Final results of the experiments will be documented in a technical report. The technical report shall receive an independent review before being submitted to NSNFP.

## **15.0 CONTROL OF ANALYTICAL SERVICES**

All anticipated analytical laboratories to be used in rendering services to this project shall be evaluated prior to providing any services. The following issues shall be addressed as a minimum:

- (a) Capability to perform project specific services;
- (b) Experience in conducting analysis requested; and
- (c) Implementation of a Quality Assurance Program that ensures quality service is performed.

A statement of work or a communication notice shall be issued to the selected laboratory to include as a minimum the following:

- a) Description of analytical services requested;
- b) Time frame for reporting the analytical results;
- c) Media in which analytical results are transmitted;

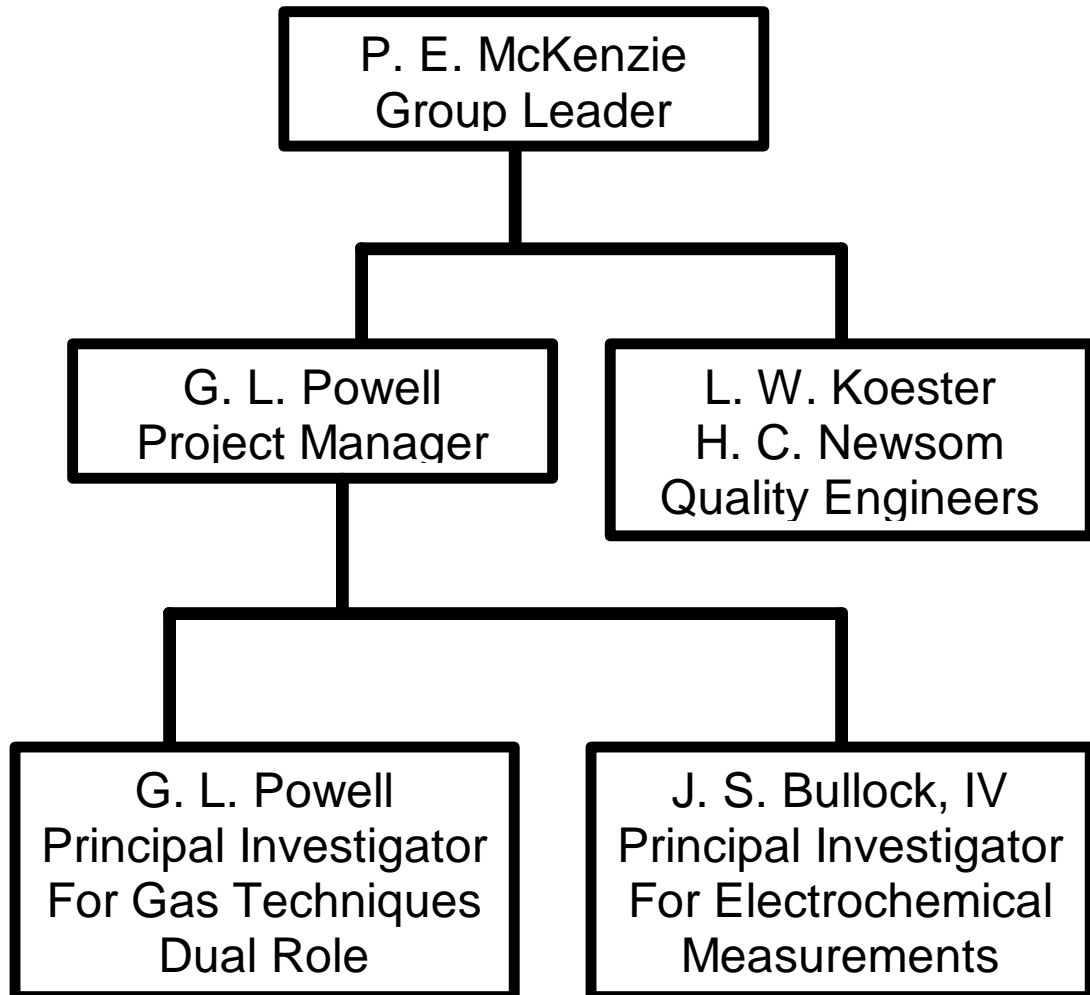


- d) Disposition of sample after analysis is complete; and
- e) Reference the QA requirements to be applied to the analysis.

Laboratory analysis reports shall be evaluated by the Project Manager, the appropriate Principal Investigator and Quality Engineer for the following:

- a) Legibility;
- b) Analysis method documented;
- c) Appropriate analysis method used;
- d) Sample identification number traceable to the results report;
- e) Personnel conducting analysis identified; and
- f) Documentation of a verification and validation process.

**APPENDIX A**  
**PROJECT ORGANIZATION**



## APPENDIX B

### DEFINITION OF WORK SCOPE AND OBJECTIVES

The project consists of conducting three separate series of related experiments, “Passivation of Uranium Hydride Powder With Oxygen and Water”, “Passivation of Uranium Hydride Powder with Surface Characterization”, and “Electrochemical Measurement of Uranium Hydride Corrosion Rate”.

The Passivation of Uranium Hydride Powder with Oxygen and Water experiment will be used for determining the rates and mechanisms of purely oxidative passivation by monitoring oxygen consumption and water production. Uranium will be hydrided inside an FTIR gas cell with a 150-mm path between ultrahigh vacuum sapphire windows. The surface area of the  $\text{UH}_3$  powder will be characterized by  $\text{H}_2$  sorption. The rate, at which the  $\text{UH}_3$  is converted from hydride to oxide by oxygen and, in a later experiment using water, will be measured. The FTIR gas analysis will be used to measure  $\text{H}_2\text{O}$  formation. Mass spectroscopy will be used to monitor  $\text{H}_2$  and  $\text{O}_2$ . The experimental measurements to be correlated are the  $\text{O}_2$  consumption rate, the  $\text{H}_2$  production rate, and the intermediate  $\text{H}_2\text{O}$  transient. This should yield reaction rates for  $\text{O}_2$  and  $\text{H}_2\text{O}$  as a function of reaction extent. This should define the effect of reaction extent on reaction rate and the fundamental reaction kinetics. The objective of this experiment is to determine the relationship between the oxidation extent and the amount of remaining uranium. As reaction extent increases along with passivation, it is expected that these measurements will be extended to elevated temperatures.

The Passivation of Uranium Hydride Powder with Surface Characterization experiment will follow the Passivation experiment. Uranium will be hydrided inside an evacuable cell with a KRS-5 window so that oxidation can be monitored by Diffuse Reflectance Fourier Transform Infrared (DRIFT) Spectroscopy, or by Emission Fourier Transform Infrared (EIFT) Spectroscopy. DRIFT will monitor the formation and stoichiometry of  $\text{UO}_{2.x}$ . EIFT will monitor thermal emission spectra during oxidation. Smaller amounts of uranium and higher  $\text{O}_2/\text{H}_2\text{O}$  levels will be used than in the gas analysis experiment. EIFT experiments will replace those originally proposed using compacted uranium hydride in metal crucibles to evaluate exothermal thermal effects of exposing  $\text{UH}_3$  powder to  $\text{O}_2$  and  $\text{H}_2\text{O}$ . The objective is to determine the quantity, stoichiometry, and exothermal effects associated with the oxidation of  $\text{UH}_3$ .

Electrochemical Measurement of Uranium Hydride Corrosion Rate approach involves the generation of a hydride layer by the electrochemical method (in situ) and the measurement, using standard computer-controlled electrochemical hardware and appropriate control and data acquisition/analysis software, of the rate of corrosion of uranium hydride surfaces. The testing scheme will use successively higher concentrations of water and oxygen and the corrosion rate will be determined for each condition. The objective of this experiment is to produce results, which, in conjunction with the gas-phase experiments, should provide a more complete picture of the oxidation phenomenon.

## APPENDIX C

### DESCRIPTION OF THE SCIENTIFIC APPROACH AND METHODOLOGY THAT WILL BE USED

#### Gas Burette Technology

The scientific approach to the gas burette technology for the formation of  $\text{UH}_3$  is described in reference 1. The FTIR gas analysis experiment will follow that technology precisely, with the infrared gas cell constituting the sample cell as described in reference 1. The use of FTIR gas analysis is given in reference 2. The DRIFT and EIFT experiments use an evacuable infrared cell with a ZnSe or KRS-5 window for the hydriding sample chamber (references 3–9). Techniques common to the use of the gas burette (references 10–15) will be used to dose the  $\text{UH}_3$  with  $\text{O}_2$ .  $\text{H}_2$  and  $\text{O}_2$  will be analyzed by dynamical mass spectroscopy using techniques for gas release measurements (references 16–19) under conditions where a small amount of the reacting gas is pulsed through the system using gas burette techniques.

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2. G. L. Powell, M. Milosevic, J. Lucania, and N. J. Harrick, "The Spectropus System, Remote Sampling Accessories for Reflectance, Emission, and Transmission Analysis using Fourier Transform Infrared Spectroscopy," *Appl. Spectrosc.* **46**, 111–125 (1992).
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## Electrochemical Experiments

The scientific approach to the electrochemical determination of  $\text{UH}_3$  corrosion rate in condensed media derives from basic electrochemical science and technology as expounded in example references 1–7. Specific experimental designs can be created, using the basic principles, to determine whatever parameters are necessary to answer the question at hand. Examples from J. S. Bullock's work of application of electrochemistry specifically to the measurement of corrosion-related phenomena are found in references 8–12. Requirements to measure corrosion phenomena in the presence of water at activities less than unity require resorting to electrolyte systems based on nonaqueous solvents with appropriate admixtures of  $\text{H}_2\text{O}$ . Such technology is described in, for example, references 13–16. The requirement to explicitly control oxygen activity over the material for purposes of determining its effect on corrosion processes calls on the understanding of its behavior contained in reference 3. The specific technical requirement for these studies is that quantitative measurement of the corrosion rate of  $\text{UH}_3$  be achieved in environments with

variable H<sub>2</sub>O and O<sub>2</sub> content. This electrochemical approach will yield corrosion current densities, which will proportionate to corrosion rates given in units of moles cm<sup>-2</sup> s<sup>-1</sup> or other equivalent units. The basic method of doing this is to perturb (polarize) the electrode potential away from its natural corrosion potential in the given environment, using externally- applied current in both the anodic and cathodic directions, and to analyze the resulting current-potential relationship to obtain the corrosion current density that is characteristic of zero external current. This is often done by linear extrapolation of the anodic and cathodic branches of the polarization semilog curve on a plot toward their mutual intersection. Since both the UH<sub>3</sub> specimens and the O<sub>2</sub> introduced into the system will be electrochemically active in the potential range associated with the corroding UH<sub>3</sub>, it will be necessary to independently take account of these partial reactions in order to separate out the actual UH<sub>3</sub> corrosion current from the measured currents. Control of H<sub>2</sub>O concentration will be done using gravimetric detection of component weights. Control of O<sub>2</sub> activity will be done using controlled dilution of O<sub>2</sub> gas using Ar; O<sub>2</sub> activity will nominally be monitored using amperometric measurement on a rotating-disk electrode operated at a potential giving a diffusion-limited current density. Other measurements such as impedance spectroscopic scans will be done using a selection of conditions to provide supplemental information to assist results interpretation.

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## **APPENDIX D**

### **APPLICABLE PROCEDURES**

#### **APPLICABLE Y-12 PLANT PROCEDURES**

1. *Quality Program Description*, Y60-101PD, Revision Date 12/15/99
2. *Control of Nonconforming Items (and Services)*, Y60-301, Revision Date 3/3/99
3. *Issues Management Program*, Y60-312, Revision Date 5/26/99
4. *Stop Work/Restart Authority*, Y60-551, Revision Date 2/12/99
5. *Procurement Quality*, Y60-701, Revision Date 3/5/99
6. *Surveillance*, Y60-904, Revision Date 4/1/99
7. *Calibration and Control of Measuring and Test Equipment (M&TE)*, Y60-802, Revision Date 1/15/99
8. *Transfer and Management of Material for Radiological Control*, Y75-101, August 4, 1998
9. *Radiological Work Permit*, Y75-122, Revision Date 2/25/99
10. *Software Quality Assurance*, ESS-QA-19.0, Revision 1 Revision Date 8/31/92
11. *Document Control*, Y15-102, Revision Date 5/26/99
12. *Chain-Of-Custody In The Analytical Services Organization*, ASO-AP-0019, Revision A

#### **APPLICABLE PROJECT SPECIFIC PROCEDURES**

1. *The Internal Calibration Procedure Process*, CRP-001
2. *Time Calibration Procedure*, CRP-002
3. *Time Synchronization Procedure*, CRP-003
4. *Volume Calibration Procedure*, CRP-004
5. *Pressure Gauge Calibration Procedure*, CRP-005
6. *Working Volume Calibration Procedure*, CRP-006
7. *FTIR Wavenumber Calibration Procedure*, CRP-007
8. *FTIR Spectrometer Setup Procedure*, CRP-008
9. *Elapsed Time Calibration Procedure*, CRP-009
10. *Mass Spectrometer Calibration Procedure*, CRP-010
11. *Gas-FTIR Background Spectrum Procedure*, CRP-011
12. *DRIFT Background Spectrum Procedure*, CRP-012
13. *EIFT Background Spectrum Procedure*, CRP-013
14. *Themocouple Calibration Procedure*, CRP-014
15. *Test Cell Reference Electrode Calibration Procedure*, CRP-015a
16. *Reference Electrode Calibration Procedure*, CRP-015
17. *Liquid Flow Calibration Procedure*, CRP-016a
18. *Gas Flow Calibration Procedure*, CRP-016b
19. *Dummy Cell Calibration Procedure*, CRP-017
20. *Resistance Measurement Calibration Procedure*, CRP-018
21. *Capacitance Measurement Calibration Procedure*, CRP-019
22. *Rotating Disk Electrode Calibration Procedure*, CRP-020
23. *pH Measurement Calibration Procedure*, CRP-021
24. *Conductivity Measurement Calibration Procedure*, CRP-022
25. *Oxygen Concentration Measurement Calibration Procedure*, CRP-023
26. *Experiment Control System Calibration Procedure*, CRP-024
27. *Voltmeter/Electrometer Calibration Procedure*, CRP-025



## **APPENDIX E**

### **EQUIPMENT AND LABORATORY APPARATUS**

#### **Equipment and Lab Apparatus**

**Principal Investigator: G. Louis Powell**

1. MKS-Baratron Pressure gauges
2. Thermocouples (Omega) New type K with Omega digital temperature readouts
3. Balances
  - Metler AE 163 M225137-sample weighings
  - Metler PE 360 (M225136) and
  - Sartorius MC1/LC 620-0 S(M-225138)-volume determination weighings
4. Bio-Rad FTIR (FTS-60 #15050) w/Harrick Scientific Spectropus System for DRIFT and EIFT
5. Nicolet 4094 Oscilloscopes
6. Mass Spectrometer (UTI 100C) with Pfeiffer-Balzers turbo molecular pump
7. Temperature Controllers, Harrick Scientific and Omega Engineering

#### **Equipment and Lab Apparatus**

**Principal Investigator: Jon S. Bullock**

1. Computer Control System, Pentium class, 133 MHz, CPU, Communicates with EC Instrumentation through IEEE 488 bus, Gateway Corp.
2. Temperature, Primary, Digital Process Indicator, Omega Model DP81, for type T thermocouple, accurate to 0.2 degrees K, Type T probes
3. Temperature, Backup, Omega Model 871A, for type K thermocouple, accurate to 0.4 degrees K, 2 units-S/Ns T-145216 and T-149171, Type K probes
4. Temperature Reference, Ice Point reference, Kaye Instruments Model K140-4A, S/N 16659
5. Peristaltic Pumps, .25% accuracy
6. Cole-Parmer Model E-03217-57 Rotameter, 2% accuracy, .25% repeatability, with NIST calibration certificate
7. General Radio Corp. Decade Resistance Standard, Model 1433X
8. Primary: Digital Micro-Ohmmeter, Valhalla Scientific Model 4300B, S/N 32-714
9. Backup: Autoranging Microvolt DMM, Kiethley Instruments Model 197, two- S/Ns 373369 and 373348

10. Fixed mica capacitor, General Radio Corp. Model 1409Y, nominal value 1.00 microfarad, accurate to 0.05% of actual calibrated value, with certificate of calibration
11. Transfer Device-Capacitance Bridge, General Radio Corp. Model 1615- A, S/N 2248, (OR DOE property #Y234973)
12. AC Impedance Dummy Cell (R-C Circuit), Model 1700-1126
13. Primary: pH Meter with ISFET Probe, Beckman Instrument Co., Model PHI 110
14. Backup: pH Meter with standard combination probe, Beckman Instrument Co. Model PHI 34
15. Alternate 1, Conductivity Meter w/glass-body cell, Jenway Co. Model PCM3, S/N 2428
16. Alternate 2, Specific Conductance Meter w/polymer-body cell, VWR Scientific Inc. Model 1054, S/N 9705006
17. Rotating Disk Electrode w/ Controller, Model AFMSRX, MSRS
18. Tachometer, Optical Retro reflective type
19. Electrolytic Test Cell: Teflon-bodied impedance-measurement cell w/annular reference electrode. Reference electrode uses H-charged Pd alloy as a hydrogen electrode. Counter electrode uses H-permeation Pd alloy helical tube.
20. Potentiostat/Galvanostat, EG&G Model 283, S/N 25101
21. Frequency Response Detector, EG&G Model 1025, S/N 12113
22. Potential Reference Half-Cell-Ag/AgCl Sure-Flow reference Electrode, Orion Research, Inc. Model 900400
23. Reference Electrode Potential Measurement- Digital Electrometer, Keithley Instruments Inc. Model 616, S/N 66428A
24. Analytical Balances, Mettler and Sartorius

## **APPENDIX F**

### **METHODS OF DOCUMENTING DATA**

Scientific (laboratory) notebooks will be used to chronicle the experiments and to both record and comment on the FTIR data and Nicolet Oscilloscope data, both of which are stored on magnetic media. The FTIR data will be transferred by diskette or Ethernet to a removable Iomega Jaz or Zip drive. The Nicolet Oscilloscope data has unique formatting on 5.25" floppy discs. They will be retained in that form with each file on a disc uniquely labeled using a time stamp of the calendar date to the nearest second so documented in the notebook. As necessary, the Nicolet data may be converted to PC formatted text and then to Microsoft Excel 97. The PC formatted text file, converted from the Nicolet format (\*.AD) will become the Nicolet record copy. Excel files will be stored with the spectroscopy data on a Jaz drive. Ultimately magnetic data will be copied to a CD-ROM as the ultimate record copy. All reporting documentation (reports, visuals, spreadsheets) shall be in the form of Microsoft Word 97 (\*.DOC), Power Point 97 (\*.PPT), Excel 97 (\*.XLS) or ASCII text E-mail (Eudora, or Microsoft Outlook).

Data will be analyzed and processed on a personal computer as magnetic media and delivered to the sponsor as both magnetic documentation and a paper copy with appropriate Y-12 release signatures.

Communications shall be by E-mail and document attachment or by magnetic media.

## **APPENDIX G**

### **CONTROL AND STORAGE OF RECORDS**

Quality assurance records are identified and categorized as a minimum into the following categories:

- a) Implementing documents
- b) Records of verification (or acceptable equivalence) of education and experience of personnel
- c) Nonconformance/deficiency reports and dispositions
- d) Laboratory notebooks
- e) Data sheets or other data records
- f) Calibration records
- g) Technical reports
- h) Records of surveillances
- i) Records of audits
- j) Records of evaluation of suppliers
- k) Records of qualification of categories C and/or D of software used for this work, reference Appendix K and Section 12.0 of this QAPjP.

All quality assurance records including magnetic data/CD-ROM data will be stored in safe # Y113393 in room 280, building 9202, Oak Ridge Y-12 Plant. This safe was purchased prior to 1967 and downgraded from storing D. O. E. classified data in 1994. Ultimately everything will be stored on CD-ROM.

## **APPENDIX H**

### **QUALITY ASSURANCE VERIFICATION OF THE WORK**

Quality assurance verification will be accomplished by surveillance of the work in progress by the Project Quality Engineer and by oversight activities performed by the National Spent Nuclear Fuel Program.

## **APPENDIX I**

### **PREREQUISITES**

- (1) This QAPjP shall be approved by the Y-12 Development Division Compatibility and Certification Group Leader.
- (2) Project personnel shall be trained to this QAPjP and implementing documents.
- (3) Resumes of major project personnel shall be reviewed against the appropriate position job description by the Project Manager and approved for project work.
- (4) M&TE shall be calibrated and documented.
- (5) Planning documents for the experiments shall be reviewed and concurred to by NSNFP.
- (6) The experiments shall be reviewed in accordance with the Y-12 Plant Development Division Integrated Safety Management System.

## APPENDIX J

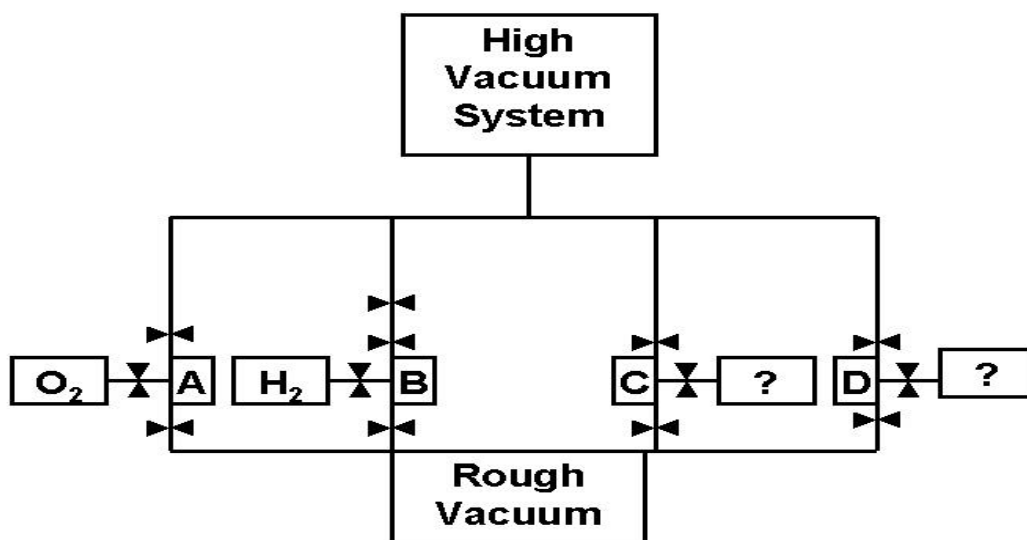
### SPECIAL CONTROLS

#### **Fire Safety Plan: Uranium Hydride Pyrophoricity (Oxidation) Studies**

The purpose of the study is to characterize the oxidation rate of uranium hydride so that the behavior of corroded uranium is understood. In this experiment uranium hydride will be prepared and reacted incrementally with oxygen to determine the mechanism by which oxidation protects uranium hydride from subsequent oxidation until the uranium hydride is mechanically disturbed. Primary concerns are that the hydrogen and the oxygen used in these experiments are of the highest purity.

The fire safety concerns is that the experiment be carried out such that hydrogen gas and oxygen gas not be mixed to form a fire/explosion hazard.

The apparatus for the experiment is shown below.



**Figure. J.1. Uranium Hydride Pyrophoricity studies apparatus.**

The experimental apparatus is a vacuum system (Turbo molecular vacuum pump, mass spectrometer, and fore pump) that services 4 modules (A, B, C, D). There is also a common roughing pump system that may operate independently of the high vacuum system. The modules have volumes that are typically 1 L or less (usually much less). The lines from the gas cylinders have 10-psi relief valves and are snubbed to guarantee that the relief valve can sustain any regulator failure. All gas lines have check valves (1/3 psi).

Manifold B supports the mass spectrometer calibration and is always connected to a hydrogen (as well as methane, carbon dioxide, carbon monoxide, and house argon). The hydrogen passes through a uranium hydride filter.

Manifold A, C, and D are experiments in which up to 10 g of uranium are converted to uranium hydride by reaction with hydrogen. Up to ~1 L STP of hydrogen is stored as uranium hydride. Up to this point we are following practices established over 20 years ago. These uranium hydride specimens will subsequently be reacted with quantities of oxygen that are small (0.1% to 10%) compared to the amount of hydrogen present as uranium hydride.

The primary safety concern is that the oxygen gas supply is inadvertently connected to the hydrogen gas supply. The following steps are proposed to prevent that situation.

The oxygen system (with 10-psi relief valve, snubbers, and check valve as described above) will not be connected to the system by removing the regulator from the bottle and capping the bottle unless the following conditions are met:

1. The hydrogen supply is disconnected from manifold B.
2. The vacuum system (both turbo and fore pump) is fully operational up to manifold B.

In future experiments, manifolds C and D may be used for similar experiments with the main difference being that the volumes and the quantities of uranium are typically 1 g.



## **APPENDIX K**

### **SOFTWARE**

#### **Software**

##### **Principal Investigator: G. Louis Powell**

1. S-Cubed Vu-Point II, Version 2.04, PC software to convert Nicolet 4094 disc data to ASCII text.
2. Bio-Rad Win-IR Version 3.01C, Level II, FTIR Operating System
3. Bio-Rad Win-IR Version 4, Level II, FTIR Operating System
4. Galactic GRAMS/32 Spectral Notebase, Version 4.01, Level I/Level II, Spectral Data Processor
5. Galactic GRAMS/32 Spectral Notebase, Version 5, Level I, Spectral Data Processor
6. Advanced Acquisition, 177-160300, Version 3.0 (88 series)/Version 3.1 (8.3A), Nicolet 4094 Program Diskettes
7. Math Pack, 177-160400 Version 3.2, Nicolet 4094 Program Diskettes
8. Martin Marietta Energy Systems (MMES), 777700, Version 2.1, Nicolet 4094 Program Disks
9. Advanced Acquisition sets the Nicolet calendar clock and time stamps the data files. Math Pack and MMES does mathematical operations on Nicolet 4094 data on the oscilloscope, Nicolet 4094 Program Diskettes.
10. Microsoft Excel 97, Spread Sheet that converts manual and text data (mostly from Nicolet 4094) for subsequent data processing.
11. Microsoft Word 97, Word Processor
12. PowerPoint 97, Visuals
13. Jandel SigmaPlot Scientific Graphing Software Versions 2.01 and version 4.01S, Data graphing
14. Jandel SigmaPlot Table Curve 2D Version 3 for Win32, Curve Fitting software
15. Nistimew, Software program that provides synchronization of computer clocks with the atomic time-scale operated by NIST.
16. Eudora electronic mail
17. Microsoft Outlook electronic mail
18. Adobe Acrobat electronic imaging.

Software is categorized in accordance with Section 12.0 of this plan as follows:

Category A: (1), (4), (5), (10), (11), (12), (13), (14), (15), (16), (17), (18)

Category B: (2), (3), (6), (7), (8), (9)

Category C: none

Category D: none

**Software (Electrochemical Specific)**

**Principal Investigator: Jon S. Bullock**

1. SoftCorr II Corrosion Software, Model 352/252, Revision 2.30, S/N USM 823, EG&G Princeton Applied Research, Corrosion Measurement Software:
2. EC Impedance Spectroscopy Software, Model PowerSine, Revision 1.0, S/N 09820312, EG&G Princeton Applied Research, Impedance Measurement
3. Equivalent Circuit, Version 4.55, University of Twente, Impedance Measurement
4. Electrochemical Impedance Software, Model 398, Revision 1.26, S/N USM 446, EG&G Princeton Applied Research, Impedance Measurement Backup
5. Equivalent Circuit, Version 4.51, University of Twente, Impedance Measurement

Software is categorized in accordance with Section 12.0 of this plan as follows:

Category A: none

Category B: (1), (2), (3), (4), (5)

Category C: none

Category D: none

## **APPENDIX L**

### **JOB DESCRIPTIONS FOR MAJOR PROJECT PARTICIPANTS**

#### **Requirements for Project Manager:**

Education: Ph. D. Degree in Chemistry

Experience: 10 years minimum experience in one or more of the following fields:

- Kinetic Spectroscopy
- Thermodynamics
- Transport Phenomena
- Isotope Effects in Metal Hydrogen
- Metal Surface Analysis
- Corrosion and Hydrogen Embrittlement

#### **Requirements for A Project Principal Investigator:**

Education: Ph. D. Degree in Chemistry

Experience: Minimum of 10 years experience in two or more of the following fields:

- Kinetic Spectroscopy
- Corrosion and Stress Corrosion
- Thermodynamics
- Electrochemical Processes
- Electrolytic Isotope Separation Processes
- Uranium Alloy Corrosion

#### **Requirements for Computational Support and Modeling Personnel:**

Education: Masters Degree (MS) in Engineering or Chemistry

Experience: Minimum of 5 years experience in 2 or more of the following fields:

- Corrosion of Uranium by Hydrogen and Water Vapor
- Modeling of Heat Transfer in Metals
- Calculation of Theoretical Infrared Spectra
- Analysis of Infrared Spectroscopic Data
- Combat Modeling With Partial Differential Equations
- Absorption of Hydrogen in Uranium
- Linear Solution for Hydriding of Uranium
- Applied Linear Analysis

#### **Requirements for the Project Quality Engineer:**

Option #1

Education: BS Degree in Engineering or related field

Experience: Minimum of 5 years experience working as a Quality Engineer, Quality Assurance Specialist, or Quality Manager

Certification: As a Quality Engineer or Quality Auditor by American Society of Quality (ASQ)

Option #2

Same as #1 with the following exceptions:

Education: AS Degree in Engineering or related field

Experience: Minimum of 10 years

## **Distribution**

### **Idaho National Engineering and Environmental Laboratory**

C. A. Dahl

### **Machine Kinetics Corporation**

H. C. Newsom

### **Oak Ridge Y-12 Plant**

M. L. Baker

J. S. Bullock

M. C. Christofferson

L. W. Koester

P. E. McKenzie

G. L. Powell

Y-12 Plant Record Services (3) [OSTI – 2, Central Files - 1]

9202 File Point

F. E. Denny